

# Ab initio structure determination by powder x-ray diffraction on Fe(2NTP)<sub>2</sub>(NCS)<sub>2</sub> complex

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## Abstract

Among the molecular switchable materials, the spin crossover (SCO) complexes have widely been studied in the last decade. In most of the cases, the SCO phenomenon appeared in Fe or Co transition metal complexes, and accompanied the changes in magnetism. In order to synthesize new SCO complexes, we synthesize two new ligands which composed of rationally designed by 5-(2-Pyridyl)-1H-Tetrazole and 1-(Bromomethyl)naphthalene. These two bidentate ligands are 2-(1-(naphthalen-2-ylmethyl)-2H-tetrazol-5-yl) pyridine (**1NTP**) and 2-(2-(naphthalen-2-ylmethyl)-2H-tetrazol-5-yl) pyridine (**2NTP**). The photoluminescence (PL) spectra of **1NTP** exhibits maximum emission intensity at 350 nm ( $\lambda_{\text{ex}} = 300$  nm), and the maximum emission intensity of **2NTP** is around 479 nm ( $\lambda_{\text{ex}} = 400$  nm). In addition, a yellow complex of Fe<sup>II</sup>(2NTP)<sub>2</sub>(NCS)<sub>2</sub> (**1**) can be obtained in powder form. Its structure is characterized by Fe K-edge extended x-ray absorption fine structure (EXAFS) and powder x-ray diffraction (PXRD). To determine the structure from PXRD, first, the cell constants are indexed from the DICVOL and N-TREOR programs. Then, based on the simulated annealing algorithm, the best structural model is obtained through global optimization in real space. In the PXRD results, complex **1** is crystallized in triclinic system. The cell constants of **1** are  $a = 14.516(7)$  Å,  $b = 14.065(8)$  Å,  $c = 8.736(8)$  Å,  $\alpha = 93.58(9)^\circ$ ,  $\beta = 98.11(8)^\circ$ , and  $\gamma = 84.95(6)^\circ$ . In the EXAF result, the coordination numbers of Fe are six, containing four N atoms at averaged distance Fe-N = 2.216(9) Å and another two N atoms of NCS ligands with averaged distance Fe-N = 2.134(9) Å. Based on the distance of Fe-N, complexes **1** is at high spin state at room temperature. Finally, the differential scanning calorimetry (DSC) measurement indicated an endothermic transition at 395 K which imply a possible phase transition happened at high temperature.

**Keywords** : Spin crossover, Powder x-ray diffraction